भारतीय मानक

IS 5282 (Part 2) : 2023

Indian Standard

गुरुत्वाकर्षण तरल अवसादन विधियों द्वारा कण आकार वितरण का निर्धारण

भाग 2 निर्धारित विंदुक पद्धति

(पहला पुनरीक्षण)

Determination of Particle Size Distribution by Gravitational Liquid Sedimentation Methods

Part 2 Fixed Pipette Method

(First Revision)

ICS 19.120

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भारतीय मानक ब्यूरो
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July 2023 **Price Group 7**

Sieves, Sieving and Other Sizing Methods Sectional Committee, CED 55

FOREWORD

This Indian Standard (Part 2) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Sieves, Sieving and Other Sizing Methods Sectional committee had been approved by the Civil Engineering Division Council.

Gravitational sedimentation particle size analysis methods are based on the settling velocity, under a gravitational field of particles in a liquid. This method is widely used for determining size distribution of many particulate materials. Typically, the gravitational methods apply to samples in the 0.5 µm to 100 µm size range and where the sedimentation condition for a Reynolds number < 0.25 is satisfied.

The choice of the most suitable method for determination of size distribution depends upon factors like:

a) the purpose for which the analysis is required,
b) the size range and density of particles,
c) the important properties of powder,
d) the amount available for test, and
e) the method by which the gross sample has been collected.

Due to the inherent characteristics of the different materials and the factor stated above, it is not possible to apply a single method of size analysis. This standard recommend procedures that may be applied in the majority of cases. The purpose of this standard is to provide uniformity in procedure for any gravitational method selected to facilitate comparisons of size analysis data.

This standard was first published in 1969 with the title 'Liquid sedimentation methods for determination of particle size of powders'. In this revision, to incorporate the latest advancement in gravitational liquid sedimentation methods for determining size distribution of particulate materials, the gravitational liquid sedimentation method has been grouped into four parts.

This standard (Part 2) describes the method to determine particle size distribution using a fixed position pipette apparatus commonly referred to as the Andreasen pipette. The Andreasen pipette employs an incremental method of analysis which gives the mass distribution directly. In incremental methods, the solids concentration at the measurement level directly determines the proportion by mass of the analysis sample that consists of particles having a diameter less than that corresponding to the velocity of fall at the time of sampling. The other parts in the series are:

Part 1 General principles and guidelines
Part 3 X-ray gravitational technique
Part 4 Balance method

In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

ISO 13317 (Part 1) Determination of particle size distribution by gravitational liquid sedimentation methods — Part 1: General principles and guidelines

ISO 13317 (Part 2) Determination of particle size distribution by gravitational liquid sedimentation methods — Part 2: Fixed pipette method

ISO 13317 (Part 3) Determination of particle size distribution by gravitational liquid sedimentation methods — Part 3: X-ray gravitational technique

ISO 13317 (Part 3) Determination of particle size distribution by gravitational liquid sedimentation methods — Part 4: Balance method

(*Continued on third cover*)

Indian Standard

DETERMINATION OF PARTICLE SIZE DISTRIBUTION BY GRAVITATIONAL LIQUID SEDIMENTATION METHODS

PART 2 FIXED PIPETTE METHOD

(First Revision)

1 SCOPE

1.1 This standard (Part 2) covers fixed pipette method for determining the particle size distributions of particulate materials, typically in the size range 1 µm to 100 µm, by gravitational sedimentation in a liquid.

1.2 The methods of determining the particle size distribution described in this standard are applicable to powders which can be dispersed in liquids or powders which are present in slurry form. The method is applicable to powders made up of particles having the same density and of comparable shape. Particles should not undergo any chemical or physical change in the suspension liquid. It is necessary that the particles have a density higher than that of the liquid.

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standard:

IS No.	*Title*
IS 4124 : 1981	Glossary of terms relating to powders (*first revision*)
IS 5282 (Part 1) : 2023	Determination of particle size distribution by gravitational liquid sedimentation methods: Part 1 General principles and guidelines (*first revision*)
IS 8883	Methods of sampling chemical and chemical product (*first revision*):
(Part 1) : 2005	General requirements and precautions
(Part 2)	Sampling equipment,
(Sec 1) : 1978	For solids
(Sec 2) : 1978	For liquids
(Sec 3) : 1978	For gases
IS 18100 : 2022	Sample preparation — Dispersing procedures for powders in liquids

3 TERMS AND DEFINITIONS

For the purpose of this standard, the definitions given in IS 4124 and IS 5282 (Part 1) shall apply.

4 SAMPLING

The sampling shall be done as the method given in IS 5282 (Part 1).

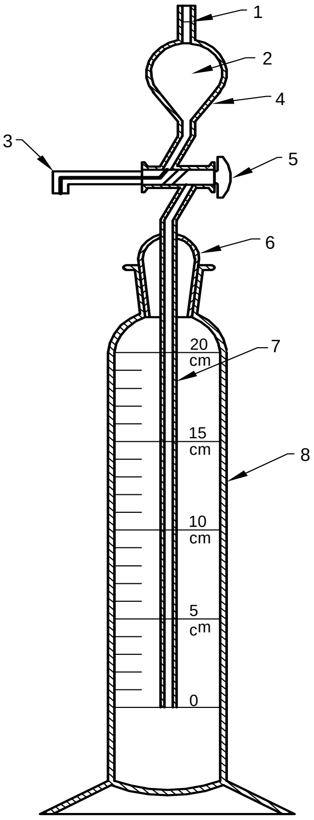
5 PRINCIPLE

In the fixed position pipette (Andresen) method, samples are withdrawn from a suspension during sedimentation by means of a calibrated pipette at a series of known times after initial agitation with the tip of the pipette being at a known depth h below the surface. After time, t, the sample withdrawn contains only those particles with Stokes diameters less than that of particles setting at rate h/t, since all particles larger than this will have settled below the sampling point. The cumulative undersize distribution by mass of the powder is obtained directly by weighing the residue after removal of the suspending medium from each extracted sample.

6 APPARATUS

6.1 Sedimentation Vessel

The sedimentation vessel is of glass of about 5 cm internal diameter and has a graduated scale from 0 cm to 20 cm marked on the side of the vessel (Fig. 1). The graduated scale may be subdivided at 5 mm or 10 mm intervals. The zero graduation should be not less than 25 mm from the inside base of the vessel so that the capacity, when filled to the 20 cm mark, is about 500 ml. It is important that the walls of the cylinder are vertical. The scale should also be vertical and have an accuracy of ± 1 mm.



Key

1	Graduation mark
2	10 ml capacity
3	Discharge tube
4	Pipette bulb
5	Two-way tap
6	Dome (vented)
7	Pipette capillary stem
8	Sedimentation vessel

FIG. 1 FIXED POSITION PIPETTE (ANDREASEN)

6.2 Pipette

The pipette is fitted with a two-way tap and a side discharge tube. The capacity of the pipette to the graduation mark is 10 ml. A bell-shaped dome (with vent hole, not shown in Fig. 1) is fused to the pipette with a ground-glass joint to fit the neck of the sedimentation vessel. The pipette bulb should be shaped as in Fig. 1. The inlet to the pipette stem should be level with the zero mark on the sedimentation vessel and the stem should be parallel to the walls of the sedimentation vessel when in position. The stem from the pipette bulb to the sampling inlet is constructed of capillary glass tube with a bore of not less than 1 mm nor more than 1.3 mm. The tube above the bulb should be a 3.5 mm bore.

NOTE — A variation of the fixed position andreasen pipette exists as the leschonski modification. In this variation, the pipette is extended to the bottom of the vessel and the sample is typically withdrawn through four apertures around the circumference of the pipette at a fixed depth of about 30 mm above the bottom of the vessel. Additionally, a subsidiary bulb with a volume matching that of the capillary is intended to remove the systematic positive error (that is, over estimation of the percentage undersize) resulting from liquid retained in the capillary tube after each withdrawal. In this way, the sample residue is removed from the capillary before taking the next sample. In practice, differences between results obtained using these modifications and results using the Andreasen pipette method may not be significant for most materials.

6.3 Ancillary Apparatus

The ancillary apparatus includes:

a) Constant temperature enclosure. For prolonged analyses (greater than 1 h), a constant temperature bath is required into which the sedimentation vessel can be immersed. It is essential that no vibration be imparted to the sedimentation vessel during the analysis (*see* **7.3**);

b) Dispersing vessel of appropriate dimensions;

c) Flexible spatula;

d) Ultrasonic bath or probe, bottle shaker or high speed mechanical stirrer;

e) Balance, having an accuracy of 0.1 mg or better;

f) Drying oven capable of being maintained within the temperature range suitable for the evaporation of the suspending liquid, for example, 378.15 K to 383.15 K for water;

g) Wide-mouthed weighing bottles or beakers suitable for evaporation, of capacity not less than 20 ml or aluminium foil containers. Aluminium foil containers, as purchased, may contain a layer of grease and should be oven dried before use. These are preferable to glass containers since they typically weigh about 1 g compared to 20 g to 30 g for the glass containers. Since a difference in mass is required, a greater accuracy can result from the use of aluminium foil containers. With certain sample systems, filtration may be preferred;

h) Desiccator in which the sample containers can be cooled after drying. Some dispersing agents are hygroscopic and the sample may absorb moisture if allowed to cool in ambient conditions; and

j) Timer with a range of at least 3 600 s and a resolution of 1 s.

7 PREPARATION

7.1 Sample Preparation

A representative sample for analysis shall be taken according to IS 8883 or any other suitable method. It should be dispersed as per IS 18100 ‘Sample preparation — Dispersing procedures for powders in liquid’ in a suspending medium of adequate viscosity. Make up the test portion to give a concentration of about 0.2 percent by volume. Record the use of ultrasonics or mechanical stirring to aid dispersion.

7.2 Calibration of Sedimentation Vessel

Determine the volume, V, of the sedimentation vessel, to an accuracy of 0.3 percent, by filling it with distilled water up to the 20 ml mark with the pipette in position. Remove typically eight 10 ml samples and determine the final height h_n after each withdrawal.

7.3 Calibration of Pipette

Clean and dry the pipette. Partly fill the sedimentation vessel with distilled water. Set the tap in the sampling position and suck water into the bulb up to the graduation mark. Turn the tap to the discharge position and allow the water to drain into a tared weighing container. Apply minimum pressure to blow any water remaining in the bulb and in the discharge tube into the weighing container. Weigh the container to the nearest 0.001 g and calculate the internal volume V_p of the pipette.

The values for the volume of the sedimentation vessel V, and the pipette volume V_p, are used to calculate the sample concentrations in the sedimentation vessel and the pipette (*see* Table 1 under Annex A). The calculated concentration using V and V_p should agree (within statistical uncertainty) with the initial concentration for samples extracted at time zero in Table 1 under Annex A.

7.4 Temperature

Maintain the temperature in accordance with IS 5282 (Part 1).

7.5 Withdrawal Time t_1 Calculation

Calculate the withdrawal time for the first fraction using Stokes law in the following form:

$$t_1 = \frac{18\eta h}{(\rho_s - \rho_l) g x_1^2}$$

where

η = Liquid viscosity, in Pa.s or mPa.s;
h = Sedimentation distance, in m;
ρ_s = Effective particle density, in kg.m^{-3} or g.cm^{-3};
g = Acceleration due to gravity, in m.s^{-2}; and
x_1 = It is the Stokes diameter for the first fraction, which must not exceed the upper critical Stokes diameter.

The initial height h is normally 20 cm, but other heights in the 10 cm to 20 cm range may be used. Extract at times calculated from selected diameters or at times in a ratio of two to one. The first sample extraction time should be not less than 60 s after the test begins.

If the size distribution extends above the coarse end of the size for the liquid, then repeat the analysis using a higher viscosity liquid until there is sufficient overlap between the two determined distributions.

8 PROCEDURE

8.1 Initial Sample Preparation

Fill the sedimentation vessel to about the 20 cm fiduciary mark (about 500 ml) with an initial homogeneous concentration of about 0.2 percent by volume. Extract 2 ml × 10 ml from the agitated suspension for drying and weighing separately as a check for the 100 percent value. It is preferable to use the pipette for this extraction so that the volumes throughout the analyses are measured with the same pipette. This sample should be dried at the same time and under the same conditions as the measurement sample, since the mass loss of the dispersing agent may depend on drying conditions. It is also advisable to dry a sample (20 ml) of the dispersing solution in order to determine whether the dried mass adsorbs moisture from air over time.

8.2 Sample Extraction Procedure

The time to fill the pipette should be about 20 s; commence extraction 10 s before the withdrawal time and terminate about 10 s after. Discharge the fraction into a tared weighing container. When the pipette bulb has drained, draw 5 ml to 7 ml of suspending liquid without dispersant into the bulb through the outlet tap.

Use vacuum suction to draw liquid into the pipette bulb. Do not use oral suction.

Remove the container whilst maintaining a slight vacuum in order to draw air into the pipette bulb, then apply pressure and wash the liquid into the tared weighing container. This removes any particles adhering to the surface. Maintain the stem of the pipette full of suspension.

8.3 Measurement Procedure

Transfer the sedimentation vessel to a constant temperature enclosure or bath and allow it to stand until temperature equilibrium has been attained, and record this temperature. When temperature equilibrium has been attained, mix the contents thoroughly by placing a finger over the vent hole in the dome and inverting continuously for 1 min.

Withdraw two samples as described in **8.2**. If it is found that the masses of these two dried samples do not agree to within 2 percent, reject the analysis. These measurements are used to determine the initial concentration of the suspension. The average of these two masses constitutes the 100 percent mass. Repeat the mixing then set the sedimentation vessel in a vertical position whilst simultaneously starting the timer. Withdraw fractions at time intervals according to **7.5**.

The depth of immersion of the pipette decreases as each fraction is withdrawn. Determine the exact decrease in depth by experiment and allow for this decrease in the subsequent calculations. For example, if the decrease is 0.40 cm for each fraction withdrawn, and at time zero the depth of immersion of the pipette was 19.2 cm, then the depth after the first timed fraction has been withdrawn will be 18.8 cm and the mean depth used for calculation of the initial diameter will be 19.0 cm. The mean depth for the second fraction will be 18.6 cm, and so on.

With some viscous liquids, a 20 s extraction time may prove insufficient and will need to be increased. If air bubbles are drawn in through the stopcock, then grease should be applied.

Repeat the sampling procedure at times in a two to one progression, for example, 2 min, 4 min, 8 min, 16 min, 32 min, 64 and perhaps 128 min, depending on the fineness of the powder. The analysis may be terminated when the extracted suspension is relatively clear.

9 ASSAY OF FRACTIONS

Accurately assay each fraction. If this is made by drying and weighing, then adopt the following procedure. Evaporate each fraction in its weighing container to dryness (as indicated by constant weight) in an oven maintained at a temperature suitable to the particular suspending liquid, and subsequently cool each container in a desiccator. Weigh the container and the dried contents to the nearest 0.1 mg and determine the mass W_n of solid material in each of the fractions (n = 1, 2, 3...). Make allowance for the mass of the dispersing agent. This is best made by drying a 20 ml sample of suspending liquid plus dispersant. With powders or dispersing agents that are hygroscopic, it is necessary to weigh without delay after removal from the desiccator. The removal of the suspending liquid may be expedited by first centrifuging the collected fractions in centrifuge tubes and decanting the supernatant liquid from the firmly compacted solid material. Then proceed as described above. Fractions may be assayed by other appropriate methods, for example, chemical or colorimetric.

10 TESTS IN DUPLICATE AND VALIDATION

10.1 Tests in Duplicate

Two test portions shall be analysed initially to determine the particle size distribution. The results are acceptable if the proportions by mass for the same Stokes diameter do not differ by more than 2 percent. If the results do not agree within 2 percent, then the analysis shall be repeated.

10.2 Validation

The checking at regular intervals of both operator procedure and instrument performance is essential to validate the test results. It is recommended to use certified reference materials for validation. A record of all validation activities shall be maintained.

11 CALCULATION OF RESULTS

11.1 Calculation of Particle Size

Calculate the Stokes diameter, x_n, corresponding to each of the corresponding heights, h_n, and time intervals, t_n (n = 1, 2, 3...).

$$x_\mathrm{n} = \left(\frac{18\eta h}{(\rho_\mathrm{s} - \rho_\mathrm{l})g^{t}n}\right)^{\frac{1}{2}}$$

11.2 Calculation of Cumulative Mass Percentage

Calculate the cumulative frequencies, F_n, by mass of particles smaller than each of the Stokes diameters, x_n, for each time interval, t_n, from the masses, W_n, of the fractions:

$$F_\mathrm{n} = 100\,\frac{W_\mathrm{n}}{W_0}$$

12 REPORTING OF RESULTS

Results will typically be presented as Stokes diameter versus cumulative distribution by mass reported to the nearest 0.1 percent. In the case of a plot, the diameters will be placed on the abscissa and the cumulative mass percentage on the ordinate. A worked-out example is given in Annex A.

The report shall state the following:

a) Sample mass and density;
b) Suspending liquid, its temperature, its density, its viscosity and, if applicable, its volume;
c) Dispersing agent and concentration of agent used;
d) Method of dispersion, including dispersing time;
e) Volume of sedimentation vessel;
f) Volume of pipette;
g) Sample concentration;
h) Instrument type used;
j) Drying of measurement samples; and
k) Any other information which could have an influence on the results.

ANNEX A

(*Clauses* 6.3 *and* 11)

WORKED-OUT EXAMPLE

Sl No. (1)	*Sample* (2)	*Quartz* (3)
i)	Sample density	2 650 $kg.m^{-3}$
ii)	Sample mass	2 205 g
iii)	Suspending liquid	Distilled water
iv)	Temperature (sample suspension)	293.15 K
v)	Liquid density	999 $kg.m^{-3}$
vi)	Liquid viscosity	1.005 mPa.s
vii)	Dispersing agent	0.1 percent (by volume) $Na_4P_2O_7$ (sodium polyphosphate)
viii)	Method of dispersion	1 min ultrasonic probe
ix)	Volume of sedimentation vessel	455 ml
x)	Volume of pipette	9.88 ml
xi)	Powder concentration	4.846 $g.I^{-1}$
xii)	Drying of measurement samples	Oven, 378.15 K
xiii)	Instrument type used	Andreasen pipette

Table 1 Worked Example (Andresen Pipette)

(*Clause* 7.3)

Sl No. (1)	**Withdrawal Time** min (2)	**Drop Height** cm (3)	**Container Mass** g (4)	**Container and Residue Mass** g (5)	**Residue Mass** g (6)	**Powder Mass** g (7)	**Stokes Diameter** µm (8)	**Cumulative Mass Undersize** percent (9)
i)	Mass of $Na_4P_2O_7$ in 10 ml		1.238 3	1.246 7	0.008 4	Average mass of $Na_4P_2O_7$ in 9.88 ml = 0.008 3 g		
ii)	0		1.237 9	1.294 4	0.056 5	0.048 0		Average 100 percent mass = (0.056 3 – 0.008 3 = 0.048 0 g
iii)	1	18.21	1.193 3	1.249 7	0.056 4	0.048 1	58.22	100.0
iv)	2	17.61	1.212 0	1.268 2	0.056 2	0.047 9	40.49	99.8
v)	4	17.01	1.171 6	1.227 7	0.056 1	0.047 8	28.14	99.6
vi)	8	16.42	1.188 1	1.240 5	0.052 4	0.044 1	19.55	91.9
vii)	16	15.82	1.222 4	1.263 7	0.041 3	0.033 0	13.57	68.7
viii)	32	15.22	1.171 5	1.193 7	0.022 2	0.013 9	9.41	28.9
ix)	64	14.62	1.220 6	1.234 3	0.013 7	0.005 4	6.52	11.2
x)	128	14.03	1.196 9	1.208 6	0.011 7	0.003 4	4.52	7.0
xi)	256	13.43	1.235 0	1.244 4	0.009 4	0.001 1	3.13	2.2

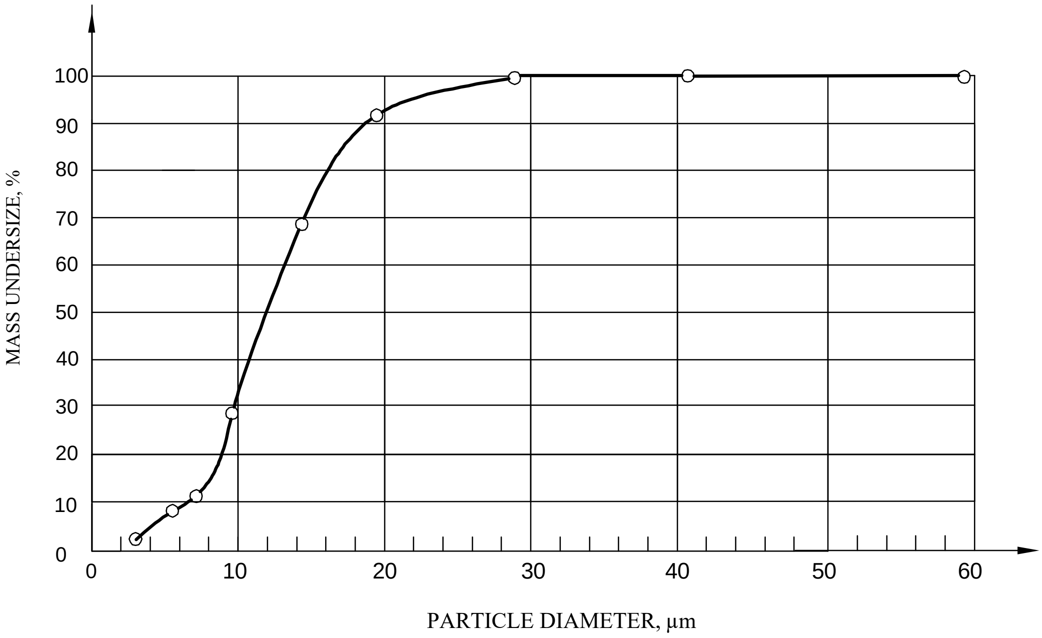


FIG. 2 CUMULATIVE MASS PERCENTAGE UNDERSIZE BY ANDREASEN PIPETTE

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Sieves, Sieving and Other Sizing Methods Sectional Committee, CED 55

Organization	*Representative(s)*
In Personal Capacity (*90, Savita Vihar, Vikas Marg, New Delhi - 110092*)	Dr R. P. Singhal **(*Chairperson*)**
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Member Secretary
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SCIENTIST 'D'/JOINT DIRECTOR
(CIVIL ENGINEERING), BIS

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The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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